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INTERNAL NOTE

EVALUATION OF POSTSOLDERING

CLEANING AGENTS

By

F. C. Osemlak

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QUALITY AND RELIABILITY ASSURANCE LABORATORY
GEORGE C. MARSHALL SPACE FLIGHT CENTER

Huntsville, Alabama

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ABSTRACT

This report presents the testing results of a selected number of postsoldering cleaning agents with respect to cleaning effectiveness, compatibility with different materials used in printed circuit assemblies, and duration of individual solvent cleaning cycles that could be tolerated without adverse effects and chemical characteristics.

The results indicate that the detrimental effects of solvents differ with the composition of the materials used in printed circuit board assemblies. The choice of cleaning agents must be specifically for the intended application, and it is necessary to perform tests on individual assemblies representative of the work to be processed.

ELECTRICAL ANALYSIS SECTION
ELECTRICAL TEST AND ANALYSIS BRANCH
ANALYTICAL OPERATIONS DIVISION

May 22, 1967

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EVALUATION OF POSTSOLDERING CLEANING AGENTS

By

F. C. Osemlak*

SUMMARY

Preliminary investigation in the evaluation of cleaning agents for the removal of flux residues and contaminants which may be present on printed circuit boards, components or assemblies after the soldering operation has indicated that this study could be most effectively pursued in two parts.

The first series of tests were selected to provide data on cleaning effectiveness of solvents. The second group of evaluations was chosen to furnish information regarding compatibility of solvents with different materials used in printed circuit assemblies and duration of individual solvent cleaning cycles that could be tolerated without degrading effects on the materials.

Consideration of these factors has shown that a "universal type solvent" is not available. Detrimental effects of solvents differ with the composition of the materials used in the printed circuit assemblies and the methods which are employed in accomplishing the removal of flux residues and contaminants. The selection of the cleaning agent is dependent upon the intended application, and it is concluded that it is essential that preliminary tests on assemblies representative of the work to be processed be performed.

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TRADE NAMES AND SYMBOLS

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MFG.	TRADE NAME OR CHEMICAL NOMENCLATURE	COMPOSITION
	Methylene Chloride	CH_2Cl_2 H_2CCl_2
	Trichloroethane	$\text{CH}_2\text{ClCHCl}_2$
	Trichlorotrifluoroethane	$\text{C Cl}_2\text{F C Cl F}_2$
Du Pont	Freon TF	Trichlorotrifluoroethane
Du Pont	Freon TA	Azeotrope of Freon TF and Acetone
Du Pont	Freon TE-35	Blend of Freon TF and Ethyl Alcohol
Du Pont	Freon TC	Azeotrope of Freon TF and Chloroform
Allied Chemical	Genesolv DTA	Azeotrope containing Trich- lorotrifluoroethane, methylene chloride and methyl alcohol
Du Pont	Freon TMC	Azeotrope containing Freon TF and Methylene Chloride
Alpha Metals	Alpha 563	Chloronated Hydrocarbon (proprietary)

SECTION I. INTRODUCTION

A. GENERAL

It has been found that the capability of a solvent to remove residues and contaminants after the soldering operation without injurious effects upon the materials of a printed circuit assembly depends upon a number of variables, among these are:

1. Soldering Flux Used. The kind and amount of flux residue remaining is dependent to a large extent upon the chemical composition of the flux used. The "activated" type of flux will usually contain, in addition to the base solvent, materials which accelerate the removal of the metal oxides and contaminants which could interfere with the making of the required solder-to-base metal bond. Whereas this removal is desirable, some of the flux residues which remain have been found to be corrosive to the electrical assemblies making it mandatory that the flux materials remaining after the soldering be removed.
2. Time Elapsed Between Soldering Operation and Flux Residue Removal. The solidification and progressive hardening of flux residue materials and other entrapped contaminants which may be present upon drying requires additional cleaning time and specific techniques for removal. The reason for this being that as the materials dry there is a shrinkage which reduces the porosity making it more difficult for the cleaning solvent to penetrate and bring the materials into solution for removal.
3. Size, Shape, and Number of Components to be Cleaned. Components containing areas with depressions, proximate to other components or to the mounting base make it more difficult to achieve the required circulation of the cleaning agent for complete removal of the residues and contaminants. These are factors that emphasize the necessity to delineate techniques of cleaning in conjunction with the choice of cleaning agent.
4. Component, Base Board, and Marking Materials. Manufacturers of electronic assemblies in practice obtain subassemblies and individual components from a number of suppliers who in turn employ multiple sources of supply. In laboratory testing, markings are completely removed on some components and show little or no effect on other

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components after cleaning with the same solvent. This effect may be extended to include base board and component materials.

5. Nature of Foreign Contaminants to be Removed. As will be discussed later, materials of different origin, organic or inorganic, may be dissolved by different types of solvents. The determination of the kinds of contaminants, other than flux residues, can be aided by a knowledge of the history of the assembly. The knowledge of storage dirt accumulation, perspiration oils due to manual handling, and lubricating compounds from material transfer equipment can assist in the choice of the cleaning agent.

6. Corrosiveness of Solvent Residue. As the degree of contamination progresses, a more vigorous type of cleaning agent is required. The limiting factor in cleaning agents employed for electronic assemblies is the immediate effects on the materials being subjected to cleaning and the possibility of attack of the materials by the solvent residue after the assembly is put to end-item usage.

From these considerations it can be seen that a "universal type solvent" is not readily determined, but that the cleaner to be used and the number of cleaning cycles and their duration will be dependent upon the individual assemblies. It is necessary to conduct tests which are specific to the application involved.

B. SOLVENT CHARACTERISTICS

Solvents may be broadly classified as being polar or nonpolar with intermediate degrees of similarity to these two classifications; therefore, it should be remembered that these terms are relative rather than absolute. In general, it may be said that polar solvents will dissolve polar materials such as acids, bases, salts, and materials ionic in nature and are associated with high dielectric constants. Chemically, polar solvents contain the hydroxy or ketonic groups i.e., alcohols, acetone. The nonpolar solvents have a low dielectric constant as typified by the hydrocarbon benzene and are more likely to dissolve nonpolar substances such as fats and oils.

Among the physical properties that are desirable in a solvent are:

- (1) Low viscosity - Allows fast drainage of parts following removal from solvent.

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- (2) Low surface tension - Permits penetration into pores, cracks, and small openings.
- (3) High density - Allows residues to be held in suspension.
- (4) Stability - Unreactive with other chemicals, particularly with oils and other hydrocarbons. Resistance to decomposition upon exposure to light and air.

Considerations of economics, toxicity, method of recovery of solvent, etc., are properly in the area of the characteristics of individual manufacturing processes to which these factors are left for evaluation.

SECTION II. OBJECTIVE

The evaluation procedure in this investigation was designed to furnish data bearing upon the following solvent cleaner characteristics:

- (1) Cleaning effectiveness.
- (2) Compatibility with materials used in printed circuit assemblies.
- (3) Duration of individual solvent cleaning cycles that could be tolerated without adverse effects.
- (4) Chemical characteristics.

To determine
a loop of
QQ-S-5
of a liquid
melted
then cleaned
followed
a micro
repeated

Flux Sample
Alpha.
100
Alpha
611
Alpha
711
Dunt
Rosin
Dut
110
Farr
#8
Ke
151
Ke
141

SECTION III. TEST PROGRAM

A. SOLVENT CLEANING EFFECTIVENESS

1. Solvent Cleaning Effectiveness of Solder Flux Residues.

To determine the cleaning effectiveness of the solvents on flux residues, a loop of solid wire solder conforming to type SN 60 WS of Specification QQ-S-571 was placed on a bright copper coupon (2" x 2") and five drops of a liquid soldering flux were added to the solder loop. The solder was melted at 205° C, the test coupon allowed to cool and the surface was then cleaned by immersing the coupon in the cleaning solvent for 30 seconds followed by rubbing with Cel-Fibe wipes. Inspection was performed with a microscope using a maximum of 20X magnification. This procedure was repeated for each solvent on all of the tested solder fluxes.

Results: Cleaning Effectiveness

Flux Sample	Solvents							
	Freon TMC	Freon TE	Genesolv DTA	Alpha 563	Freon TF	Trichloroethane	Methylene Chloride	Ethyl Alcohol
Alpha 100	A	B	B+	B	C	B+	A	B+
Alpha 611	A	B	B	B	B	A	A	B
Alpha 711	B	B+	B+	B+	C	B+	A	B
Dunton Rosin X	A	B	B	B	C	A	A	A
Dutch Boy 110	B	B	B	B	C	A	A	B
Farrelloy #8	B+	B	B+	B+	C	B	A	B
Kester 1544	B	B	A	B	C	B	A	B
Kester 135	B	B	B+	B	B	B	B	B

Rating Designations:

- A - Complete Removal of Remaining Flux.
- B+ - Removal less than 100%; more than 90%.
- B - Incomplete removal.
- C - No removal.

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2. Solvent Cleaning Effectiveness of PC Boards After

Dip-Spin Coating. Cleaning of printed circuits solder coated by the dip-spin process: Eight printed circuit boards were dip-spin solder coated on an Electrovert machine using Kester 1544 Flux, Kester 63/37 ASTM Class A solder and peanut oils at a temperature of 465° F. The boards were then subjected to a 30-second immersion with mild agitation in the solvents being tested and wiped with Cel-Fibe wipes.

B.

of solvent
mersion i

Cleaning Effectiveness	Solvents							
	Freon TMC	Freon TE	Genesolv DTA	Alpha 563	Freon TF	1, 1, 1 Trichloroethane	Methylene Chloride	Ethyl Alcohol
A-Complete Cleaning	X	X	X	X			X	
B+-More than 90% less than 100%					X	X		
B-Incomplete Cleaning								X
C-No removal of residues								
D-Surface film deposit							X	X

Solvent
Genesolv
Alpha 563
Freon TF
Freon TE
TF (not)
Ethyl Al
1, 1, 1, roethane
Methyl Chloride

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B. COMPONENT AND MATERIALS COMPATIBILITY WITH SOLVENT

1. Effects of Cleaning Agents on Formica. The effects of solvents on printed circuit board materials was determined by immersion in the cleaning solvents for varying controlled periods of time.

Formica, Grade - FF-91
NEMA - G - 10
MIL-P-18177 - C GEE

Solvent	Immersion Time								
	30 Sec.	1 Min.	3 Min.	5 Min.	10 Min.	30 Min.	1 Hr.	2 Hr.	4 Hr.
Genesolv DTA	1	1	2	2	2	2	2	2**	2**
Alpha 563	1	1	1	1	1	1	1	1	1
Freon TE	1	1	1	1	2	2	2	2	2
Freon TMC	1	1	2	2	2	2	2	2	2
TF (not mixed)	1	1	1	1	1	1	1	1	1
Ethyl Alcohol	1	1	1	1	1	1	1	1	1
1, 1, 1, Trichloroethane	1	1	1	1	1	1	1	1	1
Methylene Chloride	1	1	2	2	2	2	2**	3**	3**

** Loss of adhesion between laminations.

1. No visible effect.
2. Slight bubbling and softening.
3. Extreme softening.
4. Disintegration.

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3. Effects of Cleaning Agents on Epoxy Conformal Coating.

Epoxy - Hysol PC-12-007

Solvent	Immersion Time								
	30 Sec.	1 Min.	3 Min.	5 Min.	10 Min.	30 Min.	1 Hr.	2 Hr.	4 Hr.
Freon TF	1	1	1	1	1	1	1	1	1
Freon TMC	1	1	1	2	2	2	3	3	3
1, 1, 1, Trichloroethane	1	1	1	1	2	2	2	2	2
Alpha 563	1	1	1	1	1	1	1	1	1
Methylene Chloride	1	2	2	3	3	4	4	4	4

NOTES: Epoxy coating was applied to printed circuit board (mica board). No effect was noted on base board.

Key to Table Entries:

1. No visible effect.
2. Slight bubbling and softening.
3. Extreme softening and blistering.
4. Complete disintegration of coating.

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4. Effects of Cleaning Agents on Silicone Conformal Coating.

Silicone Slygard #182

Solvent	Immersion Time								
	30 Sec.	1 Min.	3 Min.	5 Min.	10 Min.	30 Min.	1 Hr.	2 Hr.	4 Hr.
Freon TF	3	3	4	4	4	4			
Freon TMC	4	4	4	4	4	4			
1, 1, 1, Trichloroethane	3	4	4	4	4	4			
Alpha 563	4	4	4	4	4	4			
Methylene Chloride	2	4	4	4	4	4			

NOTES: Silicone coatings were applied to printed circuit boards material (mica board).

Key to Table Entries:

1. No visible effect.
2. Slight bubbling and softening.
3. Extreme softening and blistering.
4. Complete disintegration of coating.

5. Effects of Cleaning Agents on Acrylic.

Acrylics Humiseal 1B12

Solvent	Immersion Time								
	30 Sec.	1 Min.	3 Min.	5 Min.	10 Min.	30 Min.	1 Hr.	2 Hr.	4 Hr.
Freon TF	1	1	1	1	2	2	3	3	4
Freon TMC	2	3	3	3	3	3	4	4	4
1, 1, 1, Trichloroethane	2	2	2	3	3	3	3	3	4
Alpha 563	2	2	3	3	3	3	3	4	4
Methylene Chloride	4	4	4	4	4	4	4	4	4

NOTES: Acrylics coating was applied to printed circuit board material (mica board).

No effect was noted on the board itself.

Key to Table Entries:

1. No visible effect.
2. Slight bubbling and softening.
3. Extreme softening and blistering.
4. Complete disintegration of coating.

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7. Effects of Cleaning Agents on Resistor (Carbon).

Resistors - Stock No. 215-0006056, Allen Bradley

Solvent	Immersion Time								
	30 Sec.	1 Min.	3 Min.	5 Min.	10 Min.	30 Min.	1 Hr.	2 Hr.	4 Hr.
Freon TF	1	1	1	1	1	1	1		
Freon TMC	1	2	2	3	3	3	3		
1, 1, 1, Trichloroethane	2	2	2	3	3	3	3		
Alpha 563	1	1	1	1	1	1	3		
Methylene Chloride	2	2	2	3	3	3	3		

NOTES: Resistors were completely immersed into the cleaning agent. Color coding was blistered or removed in every instance except as noted.

Key to Table Entries:

1. No visible effect.
2. Slight softening of color coding or markings.
3. Extreme softening of color coding or markings.
4. Softening of component material.
5. Complete destruction of component material.

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8. Effects of Cleaning Agents on Resistors (Film Type).

Resistors - Stock No. 215-0001739
Part No. R1 42AD152J

Solvent	Immersion Time								
	30 Sec.	1 Min.	3 Min.	5 Min.	10 Min.	30 Min.	1 Hr.	2 Hr.	4 Hr.
Freon TF	1	1	1	1	4	4	4	4	4
Freon TMC	1	1	2	4	4	4	4	4	4
1, 1, 1, Trichloroethane	1	1	2	2	4	4	4	4	4
Alpha 563	1	1	1	1	2	2	3	3	3
Methylene Chloride	2	3	3	3	3	4	5	5	5

NOTES: Resistors were completely immersed into solvent.

Key to Table Entries:

1. No visible effect.
2. Slight softening of color coding or markings.
3. Extreme softening of color coding or markings.
4. Softening of component material.
5. Complete destruction of component material.

9. Effects of Cleaning Agents on Diodes (Glass).

Diodes - Glass

Solvent	Immersion Time								
	30 Sec.	1 Min.	3 Min.	5 Min.	10 Min.	30 Min.	1 Hr.	2 Hr.	4 Hr.
Freon TF	1	2	3						
Freon TMC	3	3	3						
1, 1, 1, Trichloroethane	3	3	3						
Alpha 563	3	3	3						
Methylene Chloride	3	3	3						

NOTES: Test was not continued due to the rapid removal of color coding.

Key to Table Entries:

1. No visible effect.
2. Slight softening of color coding or markings.
3. Extreme softening of color coding or markings.
4. Softening of component material.
5. Complete destruction of component material.

10. Effects of Cleaning Agents on Diodes.

Diodes 1N470

Solvent	Immersion Time								
	30 Sec.	1 Min.	3 Min.	5 Min.	10 Min.	30 Min.	1 Hr.	2 Hr.	4 Hr.
Freon TF	1	1	1	1					
Freon TMC	2	2	2	2					
1, 1, 1, Trichloroethane	1	1	1	1					
Alpha 563	2	2	2	2					
Methylene Chloride	2	2	2	2					

NOTES: Condition of diodes employed no changes during entire length of test. No further time was spent on this diode.

Key to Table Entries:

1. No visible effect.
2. Slight softening of color coding or markings.
3. Extreme softening of color coding or markings.
4. Softening of component material.
5. Complete destruction of component material.

11. Effects of Cleaning Agents on Nylon.

Nylon Boards

Solvent	Immersion Time								
	30 Sec.	1 Min.	3 Min.	5 Min.	10 Min.	30 Min.	1 Hr.	2 Hr.	4 Hr.
Freon TF	1	1	1	1	1	1	1	1	1
Freon TMC	1	1	1	1	1	1	1	1	1
1, 1, 1, Trichloroethane	1	1	1	1	1	1	1	1	1
Alpha 563	1	1	1	1	1	1	1	1	1
Methylene Chloride	1	1	1	1	1	1	1	1	1

NOTES: Hardness Testing. All boards were tested with Shore D hardness tester. The control board checked on average of 65 Shore D. All solvents showed no effect except methylene chloride. Methylene chloride showed a very slight softening from 65 Shore D to 58 Shore D. However, no visible effect was noticed.

Key to Table Entries:

1. No visible effect.
2. Slight softening or bubbling.
3. Extreme softening or blistering.
4. Separation of material with softening and stretching.
5. Complete disintegration.

12. Effects of Cleaning Agents on Teflon.

Teflon Boards

Solvent	Immersion Time								
	30 Sec.	1 Min.	3 Min.	5 Min.	10 Min.	30 Min.	1 Hr.	2 Hr.	4 Hr.
Freon TF	1	1	1	1	1	1	1	1	1
Freon TMC	1	1	1	1	1	1	1	1	1
1, 1, 1, Trichloroethane	1	1	1	1	1	1	1	1	1
Alpha 563	1	1	1	1	1	1	1	1	1
Methylene Chloride	1	1	1	1	1	1	1	1	1

NOTES: Hardness Testing. All boards were tested with Shore D hardness tester. Control board checked on average of 73 Shore D. All solvents showed no effect.

Key to Table Entries:

1. No visible effect.
2. Slight softening or bubbling.
3. Extreme softening or blistering.
4. Separation of material with softening and stretching.
5. Complete disintegration.

13. Effects of Cleaning Agents on Semiconductor Cans.

Semiconductor 5960 - LNI - 7940P
GE - P/N-USAF-1N647

Solvent	Immersion Time								
	30 Sec.	1 Min.	3 Min.	5 Min.	10 Min.	30 Min.	1 Hr.	2 Hr.	4 Hr.
Freon TF	1	1	1	1	1	1			
Freon TMC	1	1	1	1	1	1			
1, 1, 1, Trichloroethane	1	1	1	1	1	1			
Alpha 563	1	1	1	1	1	1			
Methylene Chloride	1	1	1	1	1	1			

NOTES: No effect could be found.

Key to Table Entries:

1. No visible effect.
2. Slight softening of color coding or markings.
3. Extreme softening of color coding or markings.
4. Softening of component material.
5. Complete destruction of component material.

14. Effects of Cleaning Agents on Heat Shrinkable Tubing.

Heat Shrinkable Tubing - Scotchite 105C

Solvent	Immersion Time								
	30 Sec.	1 Min.	3 Min.	5 Min.	10 Min.	30 Min.	1 Hr.	2 Hr.	4 Hr.
Freon TF	1	1	1	1	1	1			
Freon TMC	1	1	1	1	1	1			
1, 1, 1, Trichloroethane	1	2	2	2	3	3			
Alpha 563	1	1	1	3	3	3			
Methylene Chloride	4	4	4	4	4	4			

NOTES: The heat shrinkable tubing was heated to shrinkage point. The tubing was brought to room temperature and submerged in the cleaning agents.

Methylene Chloride softened the tubing from 30 Shore D to 10 Shore D.

Key to Table Entries:

1. No visible effect.
2. Slight bubbling and softening and blistering.
3. Extreme softening and blistering.
4. Complete disintegration of material.

15. Comparison of Solvent's Effects on Hardness of Formica Board Before and After 4 Hour Immersion.

Solvents	Shore Durometer Hardness - D Scale	
	Initial (Before Immersion)	Final (After 4 Hr. Immersion)
Genesolv DTA	93	88
Methylene Chloride	93	79
1, 1, 1, Trichloroethane	93	92
Alpha 563	93	92
Freon TE	93	82
Freon TMC	93	82
Freon TF	93	91
Ethyl Alcohol	93	91

16. Acid Number Determination Performed in Accordance with MSFC-SPEC-237A.

Solvents	Acid No. KOH/GM Solvent
Freon TF	0.0034
Genesolv DTA	0.0026
Alpha 563	0.0049
Freon TMC	0.0100
Methylene Chloride	0.0043

It is not to be construed that the acid number of a solvent is the sole determinant of corrosiveness and/or detrimental effects toward all types of materials.

SECTION IV. CONCLUSIONS

A. SUMMARY

The results derived from current testing by this laboratory and a review of the literature and other laboratory data emphasizes the conclusions which are stated or implied by most investigators on the subject of postsoldering cleaning agents.

The factors to be considered in the choice of cleaning agents are:

- (1) The time of immersion of printed circuit board materials and components is critical. An immersion time of 30 seconds or less is recommended for printed circuit assemblies. This should include the total time (additive) i.e., after rework, resoldering, etc.
- (2) The materials on which the soldering is performed is to be considered. Detrimental effects of solvents differ with the composition of printed circuit boards

and the time of immersion. Cleaning agents containing Methylene Chloride are decidedly more destructive to organic materials contained in printed circuit assemblies as noted in the pure Methylene Chloride, Freon TMC, and Genesolv DTA. These solutions could not be approved without extensive tests and evaluation.

- (3) The choice of cleaning agents must be specific for the intended application. It is necessary to perform tests on individual assemblies representative of the work to be processed. Generally, if the cleaning effectiveness is satisfactory, Ethyl alcohol, Freon TF (Trichlorotrifluoroethane) or Freon TE-35 (Freon TF with 35% ethyl alcohol) should be used. Alpha 563 or a Trichloroethane may be used safely on many printed circuit assemblies and are more efficient in removing contaminants. Caution must be exercised when using pure chlorinated hydrocarbon on the following materials:

Polyurethanes
Silicones
Acrylics
Color Coding (marking).

B. OTHER CRITICAL FACTORS

- (1) The details of the method of washing and rinsing following solvent cleaning must be explicit, i.e., time of rinse, temperature of solutions, agitation, level of contamination-free solutions, etc.
- (2) Polar versus nonpolar solutions. Many proprietary solutions contain both a polar and nonpolar compound that perform satisfactorily, i.e., the nonpolar compound removes resinous materials and the polar compound removes active inorganic type halogens, etc. The active agents are usually invisible and must be removed by either rinsing or using a homogeneous stable polar and nonpolar solution. Periodic tests should be performed to ascertain the cleanliness of

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printed circuit assemblies, e.g., halogen ion test or conductivity elevation. This would also indicate the contamination level of the final rinse solution.

- (3) The marking or color coding on component parts are extremely susceptible to damage by cleaning solutions. These markings may be inks, paints, etc., with or without a protective resin cover coating. After immersion of the part into a cleaning solution, an abrasive wiping action (for drying) removes many of the coatings. Whereas, air drying without rubbing allows the markings to "reset" and retains its original adhesive properties.